Oct. 16, 1956 R. L. MAYCOCK ET AL 2,767,068

METHOD AND APPARATUS FOR CONTACTING LIQUIDS BY RECIPROCAL DISPERSION

Filed March 3, 1954 4 Sheets-Sheet 1

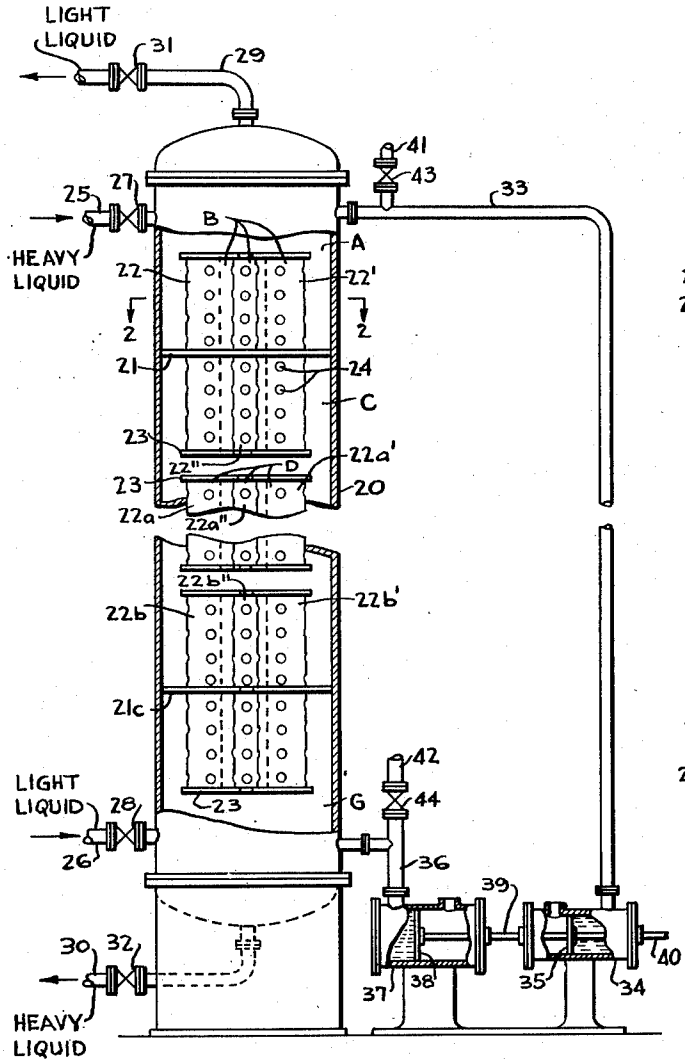


FIG. 1

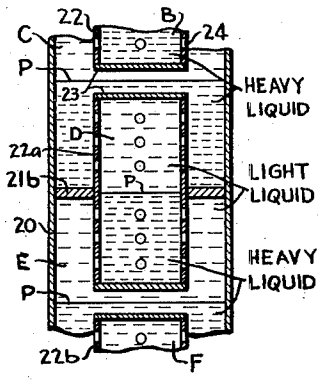


FIG. 3

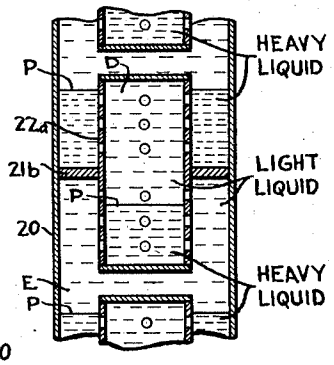


FIG. 4

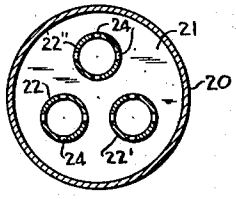


FIG. 2

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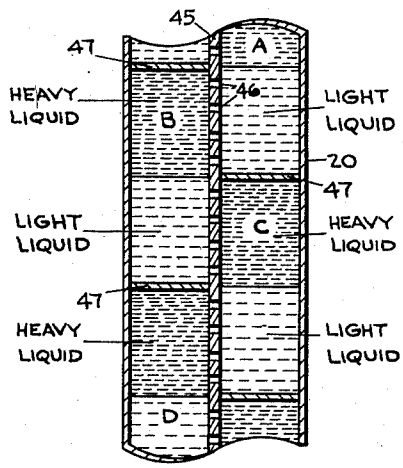


FIG. 5

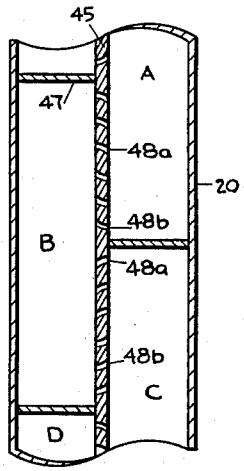


FIG. 6

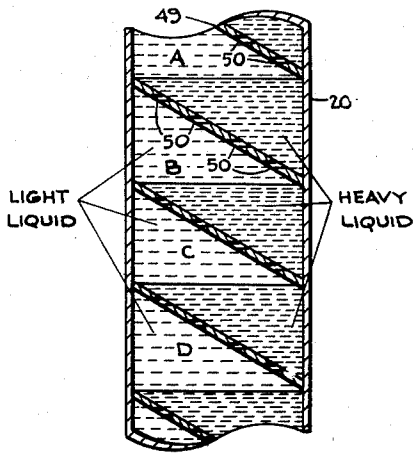


FIG. 7

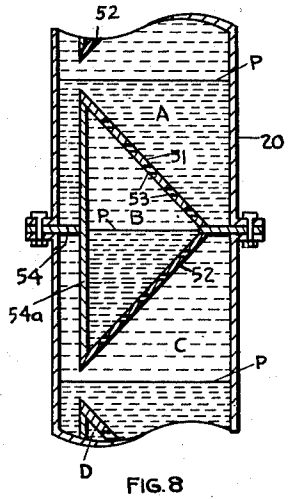


FIG. 8

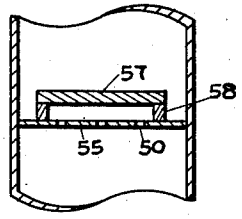


FIG. 9

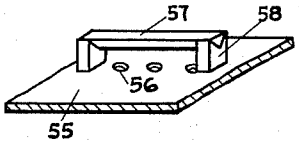


FIG. 10

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METHOD AND APPARATUS FOR CONTACTING LIQUIDS BY RECIPROCAL DISPERSION

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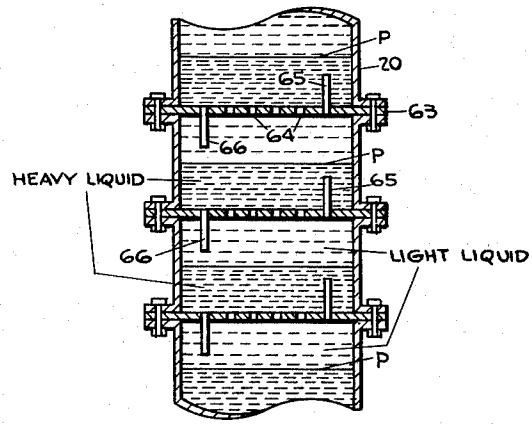


FIG. 15

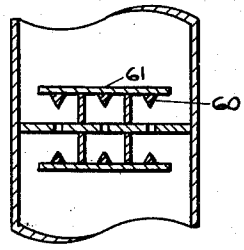


FIG. 13

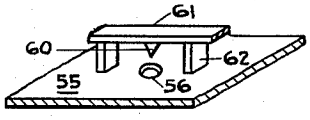


FIG. 14

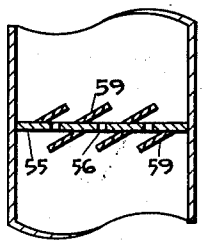


FIG. 11

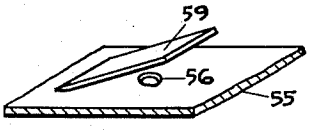


FIG. 12

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METHOD AND APPARATUS FOR CONTACTING LIQUIDS BY RECIPROCAL DISPERSION

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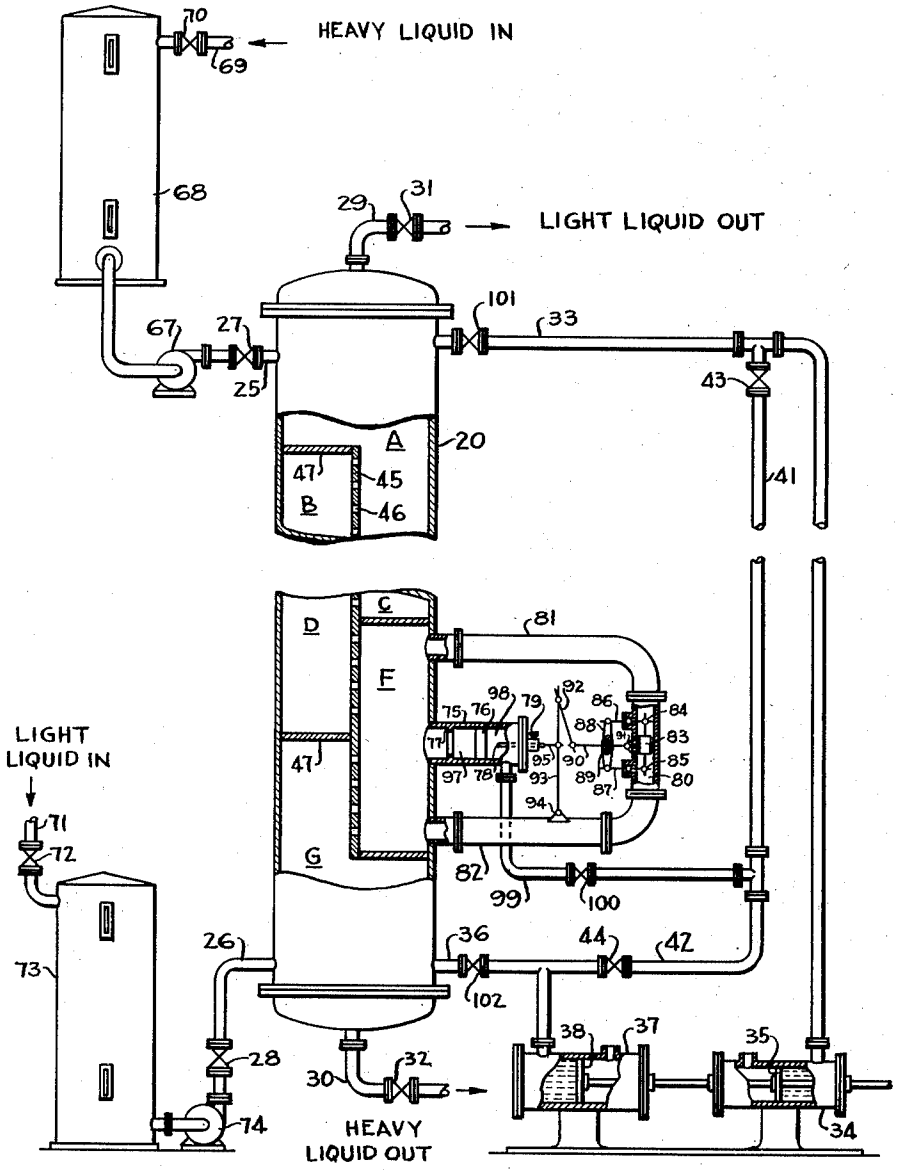


FIG. 16

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United States Patent Office 2,767,068 Patented Oct. 16, 1956

2,767,068

METHOD AND APPARATUS FOR CONTACTING LIQUIDS BY RECIPROCAL DISPERSION

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Application March 3, 1954, Serial No. 413,758

18 Claims. (Cl. 23—310)

This invention relates to a method and apparatus for intimately contacting two or more liquids forming two liquid phases having relatively different densities by reciprocal or double dispersion, i. e., by alternately (*a*) dispersing the first liquid into the second liquid and (*b*) dispersing the second liquid into the first liquid. More particularly, the invention relates to an improved method and apparatus for intimately contacting such liquids by countercurrent flow of the two liquids through a confined contacting zone bounded on two sides thereof by dispersing members, such as plates having orificial openings, by which a liquid may be introduced as a plurality of small filamentary streams into a body of the other and thereby be dispersed in the latter.

This application is a continuation-in-part of our copending application Serial No. 709,008, filed November 9, 1946, now abandoned.

It has been proposed by Van Dijck, in U. S. Patent No. 2,011,186, to improve the contacting of relatively immiscible liquids of relatively different densities by dispersing a heavier liquid downwardly through relatively small openings in a preforated plate and into an extended body of a continuous phase of a second, lighter liquid that is contiguous to the plate and thereafter effecting the reverse or reciprocal operation, wherein the lighter liquid is dispersed upwardly through the openings in such a plate and into an extended body of a continuous phase of the heavier liquid, to attain more efficient contacting, such as in an extraction process. Such a sequence of operations, wherein a liquid A is first dispersed into a continuous body of another liquid B and thereafter the liquid B is dispersed into a continuous body of the liquid A is, for brevity, herein referred to as alternately dispersing each liquid into a continuous body of the other liquid or, still more briefly, as reciprocally dispersing the liquids in each other.

The present invention is in the nature of an improvement on the above Van Dijck method and apparatus, but is not restricted to the specific mechanism or flow control operations employed for causing the alternate flow of the liquids through the dispersing members.

When the method and apparatus disclosed in the aforesaid Van Dijck patent are employed in countercurrent contacting processes, such as the solvent extraction of lubricating oils and the like, there is a change in the relative volumes of the two liquid phases within the contacting zone defined by the walls of the vessel and two consecutive perforated plates, caused by the transfer of material from one liquid phase to the other or by other causes. For example, when one liquid is oil containing several components and the other is a selective solvent for one of such components, there occurs an at least partial transfer of one or more such components from the oil into the solvent, resulting in a decrease in the volume of the oil phase and an increase in the volume of the solvent phase. This change in relative volumes is progressive as the extraction proceeds and causes a progressive change in the interface level between the

layers of the lighter and heavier liquids in each contacting zone until the thickness of one of the bodies of liquid in the contacting zone is reduced to the point at which it no longer serves as an effective continuous phase into which the other liquid may be dispersed from the other side of the perforated plate and attain a significant transfer of solute or component. When that state is reached, which may be attained after a relatively small number of cycles of the alternate dispersions have been carried out, each perforated plate becomes effective only as a one-way dispersion plate which, however, is still considerably more efficient than other methods of contacting.

The following simplified considerations will make it clear why such a state must be reached as indicated above: Consider only a single, restricted contacting zone of fixed volume defined by the walls of a vertical vessel and by an adjoining pair of transverse perforated dispersing plates extending fully across the column. This zone is filled and contains 100 volume units each of a relatively lighter and a heavier liquid, these liquids being stratified with the heavier liquid resting on the lower plate, and liquids in the neighboring contacting zones being similarly stratified so that a layer of heavier liquid in the higher adjoining zone is in contact with the upper side of the upper plate and a layer of lighter liquid in the lower adjoining zone is in contact with the lower side of the lower plate. Assume that the alternate relative motions of the plate and liquids are such as to force 50 volume units of liquid through each plate during each half of the cycle of operation. Assume further that as a result of the indicated dispersions, there is a component (solute) transfer from the lighter to the heavier liquid and that the relative volume of the lighter liquid decreases while that of the heavier liquid increases, the absolute volume changes being equal. Now, simultaneously with the dispersion of 50 volumes of lighter liquid from the said lower zone upwardly through the lower plate into the defined zone, 50 volumes of lighter liquid will be dispersed out of the defined zone upwardly through the upper perforated plate. But the dispersion of 50 volumes of the lighter liquid into the defined zone increases the light phase by less than 50 (say only by 40) volumes due to solute transfer from the lighter liquid to the heavier liquid. Thus, with an effective intake of only 40 volumes of lighter liquid and a loss of 50 volumes of this liquid by upward flow out of the zone, there is a net loss of, say 10 volumes of the lighter phase, leaving now only 90 volumes of lighter phase in said zone. Simultaneously, the volume of the heavier phase is increased and now amounts to 110 volumes. Furthermore, each subsequent dispersion, regardless of which is the dispersed phase, results in a similar transfer of solute and an additional loss of volume of lighter phase in the contacting zone. It is clear, therefore, that eventually the volume of the lighter liquid in a given restricted contacting zone at a given time will become so small that the depth of the body of lighter phase lying next to the upper perforated dispersion plate will not be sufficient for effectively contacting the heavier liquid when the latter is dispersed downwardly into said zone from the upper plate; thus, it has been found that during such downward dispersion of the heavier liquid the jets debouching from the openings pass entirely through the thin layer of lighter liquid and into the lower body of heavier liquid without any appreciable transfer of solute from one liquid to the other.

In addition to the loss of an effective body of one of the liquids, A, into which the other liquid, B, can be dispersed, the absence of a sufficient depth of one of the liquids, A, in contact with the perforated plate results in the passage of both liquids A and B through the plate during the part of the cycle when that one liquid, A, alone

should be passed therethrough. Thus, when in the example of the foregoing paragraph the volume of the lighter liquid remaining in the restricted contacting zone is less than 50 volumes, say 30 volumes, and 50 volumes of liquid flow upwardly through each dispersion plate in the course of half a cycle, all of the 30 volumes of lighter liquid are forced through the upper perforated plate early during this part of the cycle, bringing the heavier liquid into contact with the plate and resulting in the upwardly flow of 20 volumes of the latter through the perforated plate. This recycle or return flow of a portion of one of the liquid, B, into a body of the same liquid, B, in a previous zone from which it had previously been dispersed, represents a loss in capacity in two ways: First, recycling of liquid B reduces directly the net throughput thereof and, second, since the total volume of liquid forced through the plates per half-cycle is fixed, any recycle of the liquid B decreases the amount of the other liquid A that is dispersed. This recycling reduces the efficiency of contacting because of mixing of a portion of liquid B that is relatively rich in solute with the body of liquid B that is relatively lean in solute, thus undoing part of the extraction that was previously accomplished.

As already indicated, other factors than relative concentrations of a given substance which is at least partially soluble in both of the liquid phases cause similar changes in relative volumes of the two liquid phases in the restricted contacting zones and, consequently, reduce the operation of a reciprocal or double dispersion process to an effectively single dispersion operation (wherein the dispersion of one liquid in a single direction only is effective for achieving intimate contact, the alternate dispersion in the opposite direction being ineffective). Thus, temperature changes and fluctuations in the flow in the contactor may cause relative volume changes.

It is, therefore, an object of the present invention to provide an improved method and apparatus for intimately contacting two counter-flowing at least partially immiscible liquids having relatively different densities wherein each fluid is alternately and effectively dispersed into a continuous body of the other. Another object is to provide such method and apparatus for contacting two at least partially immiscible liquids by reciprocal dispersion wherein separate bodies of substantial depth of both liquids are maintained during an extended sequence of alternate dispersions. A further object is to provide apparatus for alternately dispersing two counter-flowing liquids, one into the other, simultaneously at two or more consecutive dispersing members affording restricted flow passages and means adapted to maintain substantially automatically a predetermined constant liquid-liquid interface level between the two liquids, said level being substantially intermediate the two dispersing members.

Other objects will become apparent from the following description.

In summary, the method of the present invention is an improvement in the process of flowing two at least partially immiscible, relatively lighter and heavier liquids countercurrently through a series of partitions that have flow passageways and are spaced apart to provide intervening confined contacting zones within each of which there is established a continuous body or layer of the heavier liquid in contact with the lower partition and a superposed continuous body or layer of lighter liquid in contact with the upper partition, and alternately (*a*) dispersing lighter liquid through the pasageways of each partition into the body of heavier liquid adjoining the partition in the next contacting zone toward one end of the series and (*b*) dispersing the heavier liquid through each partition through the passageways of each partition into the body of lighter liquid adjoining the partition in the next contacting zone toward the other end of the series. The improvement resides in maintaining both the continuous bodies of liquid within each confined zone at substantial depths despite changes in the relative volumes of the liquids by the step of transferring part of the liquid that undergoes an increase in volume from a given stage to the adjoining zone toward which the other liquid is dispersed only during the dispersal of the latter liquid; transfer of the stated liquid is preferably into the body of the same liquid in the said adjoining zone.

The transfer of the stated liquid is preferably effected automatically by disposing said pasageways to provide separate, simultaneous and direct communications between the said adjoining zone and a number of points distributed through a range of levels in the given stage; these passages may communicate with the said adjoining zone at a common level but will, in most embodiments, communicate therewith also with points distributed through a range of levels.

For example, in the case wherein the heavier liquid is a solvent that increases in volume by the transfer of solute from the lighter liquid, it is the heavier liquid that is transferred to a higher stage simultaneously with the dispersal of lighter liquid into the body of heavier liquid in the adjoining higher stage. With apparatus arranged as described in the preceding paragraph, the interface level between the bodies of light and heavy liquid within the given zone is below the said range of levels from which the passageways establish separate and direct communications to the adjoining higher zone when said bodies are at the proper depths within the zone; as long as this condition prevails no corrective transfer of heavier liquid is made. However, as the alternate dispersions proceed there is a small but progressive increase in the volume of the heavier liquid relative to that of the lighter liquid, and the interface rises and eventually enters the said range of levels; when this condition is reached, during the upward dispersion of lighter liquid, one or more of the passageways will transport heavier liquid upwards into the higher stage simultaneously with upward flow of lighter liquid through the other pasageways. This automatically corrects for the undesired shift in the interface level. On the subsequent downward dispersion, only heavier liquid is dispersed. It is seen that the corrective action occurs immediately upon the occurrence of any shift in the interface large enough to move the interface into the said range of levels, whereby any extensive shift in the position of the interface is prevented. This corrective transport of heavier liquid upwards may continue through one or more cycles, until the interface level is again below the said range of levels.

According to an optional variant of the invention, the above-described corrective transfer of liquid is practiced and, in addition thereto, the effective volume of one or more contacting zones is alternately increased and decreased. This is advantageously effected by decreasing the effective volume of the contacting zone during the dispersion of the liquid that tends to increase in volume (i. e., during the downward dispersion in the example of the preceding paragraph), so that the quantity of such liquid that is dispersed out of the zone through one partition exceeds the quantity of the same liquid that is simultaneously dispersed into the zone through the other partition thereof; and by increasing the effective volume of the zone during the alternate dispersion of the liquid that tends to decrease in volume, whereby the zone receives a relatively greater quantity of that liquid than is expelled simultaneously. This added step per se and the apparatus for effecting this alternate increase and decrease in the effective volume of the zone are described in detail and claimed in our copending application Serial No. 255,692, filed November 9, 1951, now Patent No. 2,728,550.

It is, of course, not essential that an exactly constant level be maintained within each compartment or contacting zone; the objects of the invention are realized when the tendency to deplete one of the continuous bodies of liquid is stopped.

The invention further provides novel apparatus within which the method may be practiced, providing a series of chambers serially connected through openings in partitions that constitute dispersing members. Thus, the apparatus may comprise an elongated, preferably upright, vessel provided with a pair of feed conduits communicating near the two ends thereof, respectively, for the liquids to be contacted, a pair of withdrawal conduits communicating near the two ends thereof, respectively for the contacted liquids, a wall structure such as a plurality of plates having orificial openings of any desired shape, e. g., circular or slot-like, for the discharge of the liquid as small drops or as filamentous jets that are disrupted into droplets by the drag imposed thereon upon flow through the continuous body of the other liquid, said dispersing members being arranged within the vessel to divide it into a plurality of compartments or chambers along its long axis, and means for causing flow of the liquids through the said openings alternately in opposite directions. Additional inlets between the ends may be provided for feeding additional liquids, and additional withdrawal lines may be similarly provided. The means for causing flow of liquid in alternate directions may be a pair of surge pumps connected to the ends of the vessel, or the partitions may be moved vertically, both being shown in the aforesaid Van Dijck Patent, No. 2,011,186, or the feed pumps that force the liquids into the terminal compartments may be used by manipuation of valves, as described hereinafter; the invention is not concerned with any specific means for effecting this alternate flow.

The particular improvement according to the invention relates to the arrangement of the passageways through the partitions between consecutive chambers. These passageways are disposed to establish separate, simultaneous and direct communication between one chamber and a plurality of points distributed through a range of levels in the adjoining chamber. In the preferred and simplest embodiment, this is achieved by positioning the partition wall to extend through different elevations (e. g., vertically or inclined) and the wall has openings distributed throughout a range of levels; it is evident that in such an embodiment the several openings or passages communicate directly with points in the first-mentioned chamber that are likewise distributed through a range of levels, whereby the apparatus is adapted to effect corrective transfer in either direction, and is therefore generally applicable regardless of whether the lighter or the heavier liquid tends to increase in volume. However, the partition may also be a horizontal plate having most openings at a common level and having an open-ended tube connected to one or more openings and extending into one of the chambers for communication therewith at a level displaced from the level of the partition. One or more similar tubes may be optionally provided on the other side of the partition to extend in the opposite vertical direction, thereby making the horizontal plate also universally applicable.

According to a specific feature of this invention, the said partitions are formed as upright tubes situated within the upright vessel, the tubes being connected to the vessel wall by transverse, imperforate partitions situated at a level intermediate the ends of the tubes, the tubes being closed at the top and bottom and having openings at a plurality of levels both above and beneath the said imperforate partitions; in this case each tube defines within itself a contacting chamber while the space outside the tubes and within the vessel wall and between each pair of imperforate partitions constitues another chamber. A particularly advantageous arrangement includes the mounting of a plurality or cluster of such tubes at a common level.

Having now described the invention in a general manner, a more detailed description will be given with reference to the accompanying drawing forming a part of this specification and showing certain preferred embodiments thereof by way of illustration, wherein:

Figure 1 is an elevation view of one embodiment of the apparatus, parts being broken away to show more detail;

Figure 2 is a transverse sectional view, taken on the line 2—2 of Figure 1;

Figure 3 is a fragmentary and diagrammatic representation of a part of the column in Figure 1 showing the desired depths of the bodies of liquid;

Figure 4 is a view like Figure 3 but showing the displacement of the liquid-liquid interface levels due to increase in the volume of the lighter liquid relatively to the heavier liquid;

Figure 5 is a fragmentary vertical sectional view of a modified construction of the apparatus having a vertical perforated plate and horizontal septa extending alternately to opposite sides of the vessel wall;

Figure 6 is a fragmentary vertical sectional view of a further modification using walls arranged as in Figure 5 but showing modified dispositions of the orificial openings;

Figure 7 is a fragmentary vertical sectional view of a further modification employing parallel, inclined, perforated plates;

Figure 8 is a fragmentary vertical sectional view of a further modification employing assemblies adapted to be bolted between sections of the vessel and using inclined perforated plates that are inclined alternately toward opposite walls of the vessel;

Figure 9 is a vertical sectional view of a perforated plate provided with dispersing means that permit the use of larger openings in the plate;

Figure 10 is a perspective view of a part of the plate of Figure 9;

Figure 11 is a vertical sectional view corresponding to Figure 9 and showing a modified dispersing means;

Figure 12 is a perspective view of a part of the plate of Figure 11;

Figure 13 is a vertical sectional view corresponding to Figure 9 and showing a further modification of a dispersing means;

Figure 14 is a perspective view of a part of the plate of Figure 13;

Figure 15 is a fragmentary vertical sectional view of a further modification of a column using horizontal perforated plates with tubes extending therefrom; and

Figure 16 is an elevation view of another embodiment of the apparatus, parts being broken away to show detail.

Referring to the drawings in detail, and particularly to Figures 1 and 2, the apparatus is designed for intimately contacting relatively heavier liquid moving downwardly through an upright column **20** with relatively lighter liquid moving upward. The column is fitted with a plurality of vertically spaced contacting assemblies, each including a horizontal, imperforate partition plate **21** that is sealed peripherally to the column wall; only two plates **21** and **21***c* and parts of two other assemblies appear in this view of the drawing, it being understood that any desired number can be mounted within the vessel. These plates have one or more, e. g., three openings for supporting a corresponding number of upright tubes **22**—**22***b*″ that extend internally unobstructed above and below the plates and are closed at the top and bottom by closure walls **23**. These tubes have perforations **24** distributed circumferentially as well as through ranges of levels situated both above and beneath the partition plates **21**. While in certain instances a single tube **22** may be mounted in each horizontal plate (see Figures 3 and 4), it is advantageous in the case of large diameter columns to mount a plurality of such as a cluster and at a common level at each horizontal imperforate partition, three such tubes **22**, **22**′ and **22**″, or **22***a*, **22***a*′ and **22***a*″, etc., being shown in the drawing. The plurality of tubes of each cluster operate in parallel and jointly constitute a contacting zone or compartment stage. Thus, the vessel is subdivided into a series of contacting chambers or compartments at progressively different elevations as follows:

The space A above the uppermost plate **21** and outside the uppermost tubes **22—22″** is the first or upper terminal compartment; the spaces B within the uppermost tubes **22—22″** jointly constitute the second stage compartments; the stage C immediately below the uppermost plate **21** and above the next plate (not shown) and outside the tubes constitutes the third chamber; the spaces D within the next cluster of tubes **22*a*—22*a″*** jointly constitute the fourth stage compartments, etc. It will be noted that the tube walls constitute vertical perforated partitions between consecutive compartments, and that the perforations **24** provide separate, simultaneous and direct connections between points situated at different elevations within one compartment and corresponding points situated at different elevations within the adjoining compartments; also each pair of consecutive compartments is vertically overlapping in that the bottom of the higher compartment is below the top of the lower compartment. The terminal compartments A and G serve as settling zones as well as contacting zones.

The terminal compartments A and G communicate with feed conduits **25** and **26**, respectively, having valves **27** and **28**, and with withdrawal conduits **29** and **30**, respectively, having valves **31** and **32**. The compartment A further communicates through a conduit **33** with the cylinder of a reciprocating surge pump **34** having a movable piston **35**, while the compartment G communicates through a conduit **36** to the cylinder of a reciprocating surge pump **37** having a movable piston **38**. The pistons are connected at **39** and are driven by a common drive means (not shown) by a shaft **40**. The pistons are thus seen to move in unison and to have the same strokes, but since the pistons are oppositely disposed, the pumps operate 180° out of phase with each other. The cross-sectional areas of the pump cylinders may be made the same or different, as shown, in accordance with the desired relation between the volumes of liquid displaced thereby. Branch pipes **41** and **42**, having normally closed valves **43** and **44**, may be provided for venting the lines, for servicing or for operating a mechanism by which the effective volumes of one or more compartments are altered, as described hereinafter for Figure 16.

The operation of the apparatus is as follows: Heavy and light liquids to be contacted are supplied through the conduits **25** and **26**, respectively, and within each of the several compartments A, B, etc., there is established a layer of heavier liquid and a layer of lighter liquid. When the column is filled the introduction of light and heavy liquids is preferably intermittent, as described herein, although continuous admission may be used; starting with both pistons of the surge pumps at the left as shown, valves **28** and **32** are open, valves **27** and **31** are closed. During the first half-cycle, lighter liquid is admitted through the conduit **26** into the bottom of the column while the surge pumps are moved toward the right; the latter causes liquid to be forced into the top of the column via the conduit **33** and liquid to be withdrawn from the bottom of the column via conduit **36**. Simultaneously, contacted heavy liquid is discharged through the conduit **30**. The effect of the surge pumps is to cause liquid to flow downwards through the column. In this flow heavier liquid is forced out from the bottom of each of the compartments A, B, C, etc., through the perforations **24** and is dispersed into the body of lighter liquid in the adjoining, lower compartment for a short time, e. g., from a quarter of a minute to five minutes, the rate of movement of the surge pump pistons being such as to complete one stroke in this period. The dispersed drops of heavier liquid settle through the continuous bodies of lighter liquid and augment the bodies of heavier liquids in the respective compartments. During the second half-cycle, the valves **27** and **31** are open, the valves **28** and **32** are closed, and the pistons move toward the left while heavier liquid is admitted through the conduit **25** and contacted lighter liquid is withdrawn via conduit **29**. The pistons cause liquid to be forced into the bottom of the column via conduit **36** and liquid to be withdrawn via conduit **33**, thereby causing lighter liquid to be forced out from the top of each compartment through the same perforations **24** and to be dispersed into the continuous body of heavier liquid in the adjoining, higher compartment for a short time, usually equal to that of the first half-cycle. The dispersed drops of lighter liquid settle upwards to augment the bodies of lighter liquid in the respective compartments. When this action is completed another complete cycle is begun. It will be understood that the valves **27, 28, 31** and **32** may be operated automatically, e. g., by cam mechanisms, not shown, in synchronism with the movements of the pump pistons.

The above preferred operation has the advantage that the feed liquids can be supplied at moderately low pressures; however, it is also possible to supply them at other times, e. g., the lighter liquid may be supplied while the pistons move toward the left and the heavier liquid supplied while the pistons move toward the right, provided appropriate supply pressures are available, or these liquids may be supplied continuously. Similarly, the contacted liquids may be withdrawn at other times, or continuously.

It may be noted that when a surge pump takes suction it draws liquid from one of the end compartments. It is not important whether the lighter or heavier liquid is drawn in; however, it is preferred to connect the conduits **33** and **36** near the extremities of the column, as shown, beyond the interface levels in the terminal compartments, whereby the conduit **33** receives settled lighter liquid and the conduit **36** receives settled heavier liquid. As regards the strokes of the pistons, it is preferred to move them gradually and slowly, so as to extend the strokes throughout the respective half-cycles; but it is also possible to move them more rapidly, so as to complete the stroke during an early part of the half-cycle, e. g., at the beginning, prior to, or during the early part of the time that feed liquid is supplied, or at the end, during the last part of or subsequent to the time that feed liquid is supplied to the column.

Often the settling of the dispersions formed in one half-cycle is not completed by the time the subsequent half-cycle is begun, and minor amounts of very small droplets of each liquid that are slow in settling are still in suspension in the other liquid. Hence, a minor amount of lighter liquid is re-dispersed downwardly through the openings **24** of the perforated partitions with the heavier liquid while a minor amount of heavier liquid is re-dispersed upwardly with the lighter liquid. To compensate for this recycling it is desirable to design the sizes of the surge pumps to flow through the perforated partitions during each half-cycle a greater volume of liquid than the net volume of the same liquid that traverses the perforated partitions in the course of a complete cycle. The net volume of liquid traversing a partition is approximately equal to the volume thereof admitted by the feed conduits.

In an operation wherein no change in the relative volumes of the two liquids occurs as they flow through the column, certain simple relationships exist between the volumes. For instance, during the described first half-cycle, if the volume of liquid entering the chamber A from the surge pump **34** is H, then the volume of heavier liquid dispersed from each compartment to a lower compartment between effective partitions (considering the several tubes of the same cluster collectively as one compartment and as defining two effective partitions, one above and the other below the imperforate plate **21**) is H. Thus, the volumes of heavier liquid gained and lost by each compartment (between effective partitions) are the same and, therefore, the liquid-liquid interfaces within each compartment remain constant at the completion of each cycle. This relation is also true in the other half cycle of operation, wherein a volume L of liquid enters the chamber G from the surge pump

37 and equal volumes L of lighter liquid flow through the several effective partitions.

The action of the apparatus in compensating for a change in the relative volumes of the liquids will now be described with reference to Figures 3 and 4, which show portions of the column, but wherein only one tube is shown at each imperforate partition plate. Figure 3 shows only chambers B, C, D, E and F, and illustrates the desired positions of the liquids, such as would result from the condition considered in the preceding paragraph. The depths of the liquid bodies are approximately equal within the several compartments, and the interface levels P are situated so that each effective perforated partition has only heavier liquid on one side and lighter liquid on the other side. Now consider the situation wherein the heavier liquid entering the top of the column contains a solute which can transfer to the lighter liquid, causing the volume of the latter to increase. Assume, for example, that ten volumes of composite heavier liquid is dispersed into the contacting zone defined by chamber D from the chamber C through the perforated partition defined by the upper part of the tube 22*a*, with resultant transfer of one volume of solute to the lighter phase; this leaves a net intake of only nine volumes of heavier liquid into the chamber D. Simultaneously, ten volumes of heavier liquid are dispersed out from the bottom of the chamber D into the chamber E. The net effect is that the volume of lighter liquid has increased by one volume while that of the heavier liquid has decreased by the same amount. On the next half-cycle, if ten volumes of lighter liquid are dispersed upwardly from the chamber E into the chamber D, additional transfer of solute occurs, with a further diminution of the volume of the heavier liquid by, say, one volume and an equal increase in the volume of the lighter liquid; simultaneously, ten volumes of lighter liquid are dispersed from the chamber D into the chamber C. Upon completion of the cycle, there has been a net increase in the volume of the lighter liquid by two volumes, and a net decrease in the volume of the heavier liquid by two volumes, whereby the interface level P is depressed as shown in Figure 4. This action occurs in all of the several contacting zones, although not necessarily to the same extent, and Figure 4 shows the interface levels P depresed also in the adjoining chambers, but by smaller distances.

It is readily seen that if no provision were made to compensate for this shift in the interface level, after an extended sequence of cycles the layer of heavier phase would assume a very thin depth. In prior art methods and apparatus, wherein horizontal perforated plates are used, this results in ineffective contacting during dispersion of lighter liquid because the lighter liquid jets through a shallow layer of heavier liquid and enters the layer of lighter liquid without effective contact with the heavier liquid. The result is that only the downward dispersions of heavier liquid are effective, and the contacting efficiency is materially reduced. Moreover, when the layer of heavier liquid is thin it is rapidly exhausted during the downward dispersion.

Now by the construction according to the invention, no more than a minor shift in the interface level can occur. If, for example, the interface P in the chamber D falls to the level of the highest holes 24 of the lower part of the tube, as shown in Figure 4, lighter liquid will be transported through those holes into the lower stage E simultaneously with the dispersal of heavier liquid through the other holes from chamber D into chamber E. This downward transport occurs only during the dispersal of the heavy liquid, which tends to decrease in volume in relation to the other liquid; on the alternate dispersion of lighter liquid upwards, all holes pass only lighter liquid. As a result of the downward transport of the lighter liquid, the depth of the layer of lighter liquid within chamber D is decreased, while that of the layer of heavier liquid is increased, thereby restoring the interface level P toward the position shown in Figure 3. While the transport of lighter liquid toward a lower stage represents a small loss in efficiency, it prevents the far greater loss due to the change from an effective double or reciprocal dispersion system to a single dispersion system, as noted above. Furthermore, the transport method of maintaining the interface level at the desired height and, consequently, the depths of the continuous bodies of liquids is automatic and requires no external control.

If, for any reason, a greater shift in interface level occurs such as to bring lighter liquid opposite additional holes below the highest holes of the lower part of the tube, additional holes come into play during the corrective flow and correspondingly larger quantities of lighter liquid are transported to the lower stage. Hence, the construction automatically varies the quantity of lighter liquid that is transported during the dispersion of the heavier liquid.

It is evident that a similar corrective action occurs when the interface level rises, e. g., due to an increase in the volume of the heavier liquid relative to that of the lighter liquid; in this event heavier liquid is transported to a higher stage during the dispersion of lighter liquid to the adjoining higher compartment, the heavier liquid flowing through the lowermost holes of the upper part of the tube and the lighter liquid flowing through the other holes. Only heavier liquid is dispersed through all holes during the alternate dispersions into consecutive lower compartments.

Example

The utility and advantage of the method and apparatus are demonstrated by the data shown in the table below. A contactor of the fixed-plate type as described in the Van Dijck Patent No. 2,011,186, was operated, starting with layers of equal thickness in each compartment and operating with alternate (dual) dispersion, as described above. After a short period of time the thickness of the upper layer of light phase decreased so that its ratio to the thickness of the heavy phase was about one-tenth. In a comparative experiment, the ratio of the thicknesses of the layers was held at about unity; operational variables other than this ratio were maintained the same. The results were:

Ratio of Light to Heavy Phase	Thickness of Layer of Light Phase	Efficiency of Four-Plate Contractor	
		Theoretical Stages	Percent Eff.
about 0.1	about 0.2 in.	2.5	63
about 1.0	about 1.0 in.	3.8	95

The results shown in the table demonstrate that in order to secure the benefits of the two-phase reciprocal dispersion, it is necessary to operate the contactor in such a manner as to maintain the ratio of the thicknesses of the two liquid phases at a value not greatly different from one (say from a ratio of about 1:4 to a ratio of about 4:1). It has been found that this ratio should preferably be maintained between about 1:2 and 2:1, unless some liquid property, such as excessive viscosity of one phase, dictates otherwise. Preferably, the minimum thickness of each layer should be at least three inches, although the data in the foregoing table indicate that thicknesses as low as one inch can be used; the exact optimum value is dependent upon such factors as the properties of the dispersed phase, including viscosity, surface tension, and the like, which properties affect the rate of drop formation after travel through the orificial opening, the jet velocity of the stream, the rate of transfer of solute from one liquid to the other, etc.

The perforated tubes 22 of Figures 1–4 may be constructed of any material which is not reactive with the liquids to be contacted and which has the required struc-

tural characteristics, such as rigidity. Thus, they may be made of glass or metal. The tubes may vary in size depending upon the size of the installation, fluid characteristics of the system to be contacted, etc. It has been found that, in general, tubes in the neighborhood of about two to four inches in diameter and from about eighteen inches to about twenty-four inches in length are satisfactory for commercial-sized columns. The number of tubes in each plate assembly may be varied to suit the individual capacity requirement very much as one varies the size of a bubble plate for a fractionating column. The number of plate assemblies may also be varied to suit the requirements of the process. The perforations should each be of such size and number as to give the desired quantity and type of dispersion. Perforations of the order of about twenty-five to seventy-five thousandths of an inch diameter have been found to be suitable for systems of the character of methyl isobutyl ketone-water-acetic acid. The spacing of the perforations may be varied in many ways; thus, it has been found advantageous to arrange the perforations so that a relative small number of them will be involved in the corrective transport of the liquid that tends to increase in volume. With larger size openings, suitable dispersing action may be obtained by a corresponding increase in the jet velocity, i. e., by an increase in the driving pressure.

The wall structure subdividing the vessel **20** into chambers may have other arrangements, as shown in Figures 5–15.

Referring to Figure 5, the column **20** contains a vertical partition plate **45** having perforations **46** and sealed along its vertical margins to the opposite walls of the column. The partition is further connected to the column by horizontal septum plates **47** that are mounted alternately on opposite sides of the partition and are sealed thereto and to the column wall, so as to define a series of chambers A, B, C, etc., that communicate with one another only through the perforations **46**. The operation of this column is as previously described; as shown in this view, each compartment is filled with a layer of heavier liquid and a layer of lighter liquid, the thicknesses of these layers being shown to be equal. Should any shift in the interface level occur, corrective action occurs as was described above.

Figure 6 shows a construction similar to that of Figure 5 but differing in that the perforations **48***a*, **48***b* in the partition **45** are inclined so that the ends of the passages communicating with the higher chamber and lower than the other ends; thus, the termini of the holes connecting chambers A and B are higher toward the latter chamber. Moreover, if desired the perforations may be curved so that their sections in a vertical plane are convex toward the nearest horizontal septum; thus, the upper perforations **48***a* in each effective plate are upwardly convex and the lower perforations **48***b* are curved convexly downwards.

Figures 7 and 8 show variants employing inclined perforated plates. In the former, the plates **49** are parallel and inclined between the horizontal and vertical and have perforations **50** distributed through different levels within each plate. The plates are sealed to the column wall and define chambers A, B, C, D, etc. In Figure 8, the contacting assemblies are adapted to be bolted between flanged sections of the column **20** and include a pair of oppositely inclined plates **51**, **52**, each having perforations **53** distributed throughout different levels of each plate. Each assembly further includes a vertical imperforate plate **54***a* and a horizontal septum plate **54** that is clamped between the column sections. This structure divides the column into the compartments A–D, shown. The operation of these columns is at that previously described.

The relatively small openings used in the partitions of Figures 1–8, as well as in Figures 15 and 16 described below, may be replaced with larger openings which offer less resistance to fluid flow and are less likely to become fouled by solid foreign matter. In order to effect the desired dispersing action when using perforated plates with larger openings, the alternating pressures delivered by the surge pumps may be suitably increased and/or deflecting surfaces may be placed near the openings to cooperate with the openings and produce a dispersing effect. Various shapes may be employed. As shown in Figures 9 and 10, the plate **55** may have large openings **56** and a wedge-shaped deflector bar **57** secured by supports **58**. In Figures **11** and **12**, the plate carries inclined deflectors **59** on both sides of the openings **56**, while in Figures 13 and 14 the plate carries a conical deflector **60** opposite each side of each opening **56**, mounted on a bar **61** and supports **62**. Although these perforated partitions were shown to be horizontal, the deflectors may be applied to the vertical or inclined partitions previously described.

When horizontal perforated plates are used, the requirement that different points distributed through a range of elevations in one chamber be simultaneously, separately and directly connected to the adjoining chamber is met by providing one or more transport tubes; such tubes may extend upwards from the plate for downward corrective transport of lighter liquid in the event that the lighter liquid tends to increase in volume, or may extend downwards from the plate for upward corrective transport of heavier liquid in the event that the heavier liquid tends to increase in volume. In the embodiment shown in Figure 15, both upwardly and downwardly extending tubes are provided, making the device useful for either type of corrective transport.

In Figure 15, the column **20** is provided with horizontal partition plates **63** having perforations **64** similar to the apparatus of the Van Dijck Patent No. 2,011,186. However, the plates carry transport tubes **65** extending upwards from each plate and also transport tubes **66** extending downwards from each plate; as was noted above, either the tubes **65** or the tubes **66** may be omitted, depending upon the nature of the liquids to be contacted. These tubes are open at both ends, the ends nearest the plate being mounted at an opening so as to communicate with the chamber on the opposite side of the plate. The open ends of the tube away from the plate are shown to be slightly less than half-way toward the next plate, so as to be immersed in the body of liquid that wets the plate on which the tube is mounted. The tubes are of restricted sizes, or contain flow restrictive devices, such that they offer about the same resistance to the flow of liquids as other perforations.

In operation, so long as the interface levels P are correct, as illustrated, the perforations and all tubes function to carry and disperse lighter liquid upwards during the half-cycles wherein the surge pumps force liquid into the bottom of the column and withdraw it at the top, and heavier liquid downwards during the alternate half-cycles, as contemplated by the Van Dijck patent. Hence the tubes **65** and **66** serve merely to pass additional liquid of the same kind as is passed by the perforations, both on the upward and downward dispersals. If, however, the volume of lighter liquid increases and the interface level P in any chamber falls until the lighter liquid contacts the upper, open end of a tube **65**, this tube transports lighter liquid into the adjacent lower chamber simultaneously with the downward dispersal of heavier liquid (through the perforations **64**) into said adjacent compartment on one stroke of the surge pumps; during the opposite stroke of the surge pumps, all perforations and tubes carry lighter liquid upwards. The net effect is that the interface level in the stated chamber is raised. Similarly, if the volume of the heavier liquid increases and the interface level in any chamber rises until the lower, open end of a tube **66** is contacted, heavier liquid is transported upwards into the adjacent higher chamber simultaneously with the upward dispersal of lighter liquid (through the perforations **64**) into said adjacent compartment on one stroke of the surge pumps; during the op-

posite stroke of the surge pumps, all perforations and tubes carry heavier liquid downwards.

Figure 16 shows a variant of the apparatus wherein means are provided for alternately increasing and decreasing the effective volume of a chamber defining a contacting zone; it further illustrates a variant for causing flow of liquids through the perforations alternately in opposite directions without recourse to surge pumps, although surge pumps are shown as one possible means. In this view the column **20** is shown to contain the wall structure previously described for Figure 5 and have feed and discharge conduits and surge pumps as described for Figure 1, all bearing corresponding reference numbers. The heavy liquid feed conduit **25** is connected to a pump **67** taking suction from a measuring tank **68** to which feed liquid is supplied through a supply conduit **69** and valve **70** from a source, not shown. Similarly, light liquid is supplied through a supply conduit **71**, valve **72**, measuring tank **73** and pump **74** to the conduit **26**.

One or more intermediate compartments, e. g., the compartment F, is formed as an expansible chamber situated in part within the column **20** and in part outside of it; the latter part is enclosed in a cylinder **75** that communicates with the interior of the column through an opening in the column wall at an intermediate level of the compartment and is provided with a floating piston **76** which thus forms a movable wall of the respective compartment F, constituting a means for altering the effective volume of the compartment or contacting zone. A fixed stop member **77** projecting inwardly from the wall of the cylinder limits the inward movement of the piston **76** (in the direction which decreases the volume of the compartment). The cylinder is provided with an adjustable stop member **78** which may be adjusted in any of various ways, either manually or automatically, to limit the outward movement of the piston **76** at desired displacements thereof. Thus, the stop may be adjusted manually at any axial position and clamped thereby set screws **79**.

The adjustable stop **78** is preferably actuated automaticaly by any suitable positioning mechanism controlled by a liquid-liquid interface level-responsive device that measures the apparent liquid-liquid interface in the contacting zone with which the stop is associated, thereby automatically varying the piston displacement from time to time as required. A level-responsive device and stop-positioning mechanism are diagrammatically shown herein and are merely indicative of known level-responsive and positioning devices that may be used. In the embodiment shown, a float chamber **80** is connected to the upper and lower parts of the compartment F by large conduits **81** and **82** and contains a float **83** that has a buoyancy to assume a position at the interface between the lighter and heavier liquids. The float is pivotally connected at **84** and **85** to levers **86** and **87** that extend out of the float chamber and are fulcrumed in the wall of the chamber and sealed thereto against leakage of liquid. The outer ends of the levers are pivotally connected to a link **88** having an intermediate notch **89** that provides a pair of abutments for moving a rod **90** up or down about a fixed pivot **91**. The free end of rod **90** is pivotally connected to one end of a link **92**, the other end of which is pivotally connected to the movable end of a second link **93** having a fixed pivot **94**. A third link **95** is pivotally connected between the stop **78** and the link **92**. It is evident that when the interface level in the chamber **80** rises the float **83** also rises, thereby depressing the link **88** and rotating the rod **90** counter-clockwise. This rotates the link **93** in a clockwise direction and moves the stop member **78** to the right, to increase the displacement of the piston **76**. Similarly, a drop in the interface level causes the stop member **78** to be moved to the left. This specific relation between the direction of the stop movement and the change in the interface level is intended for the situation described below, wherein the volume of the relatively lighter liquid tends to decrease in relation to that of the heavier liquid; when the volume of the lighter liquid tends to increase in relation to that of the heavier liquid the linkage is arranged to move the stop **78** to the left when the interface level rises.

It will be noted that the outer end of the cylinder **75** is closed and that the piston **76** divides the cylinder into inner and outer spaces **97** and **98**, respectively. The outer space **98** is connected by a conduit **99** and valve **100** to means for alternately forcing fluid into the space **98** and withdrawing fluid therefrom. For example, the conduit **99** may be connected to the conduits **33** or **36** by the conduits **41** and **42**, respectively. The conduits **33** and **36** may be provided with shut-off valves **101** and **102**.

In operation, only one of the valves **43** and **44** is open and the other is closed, while the valve **100** is normally open. The selection of the connection of the conduit **99** depends on the direction in which the liquid-liquid interface tends to move due to changes in the relative volumes of the liquid phases. In the example to be described, wherein the lighter liquid tends to decrease in volume relatively to the heavier liquid tending toward a rise in the interface level, the valve **43** is opened, placing each space **98** into communication with the surge pump **34**, and the valve **44** is closed; when the relative volume change occurs in the opposite sense, the valve **43** is closed and valve **44** is opened, placing the space **98** into communication with the surge pump **38**. When the pressure in the conduit **99** and space **98** exceeds the pressure in the contacting zone F the floating piston **76** is moved leftward to the positions determined by the stops **77** thereby decreasing the effective volume of the zone; when the pressure in the space **98** is less than that in the contacting zone the piston moves rightward until it engages the stops **78**. Thus, the floating piston is actuated by fluid pressure.

As employed, for example, to extract a petroleum hydrocarbon fraction with a selective solvent having a higher density, such as furfural, these liquids are admitted through the inlet conduits **71** and **69**, respectively. They flow countercurrently through the column **20** until each contacting zone contains some of each of these liquids. In each zone the heavier liquid settles to the bottom and the lighter liquid rises to the top, so that there is a body of substantial thickness of liquid phase consisting substantially only of the heavier liquid on the upper side of each plate **47** and a body of substantial thickness of liquid phase consisting substantially only of the lighter liquid in contact with the lower side of each plate; this is shown in Fig. 5. The openings **46** in the perforated plate are of such dimensions as to result in flow resistance great enough to prevent any appreciable liquid movement through the openings caused only by the difference in specific gravities of the liquids. Now the two counterflowing liquids are alternately dispersed, one into the other, by forcing the liquids through the openings **46** alternately in opposite directions, and contacted lighter and heavier liquids are withdrawn through the conduits **29** and **30**, respectively. Several modes of operation are possible and will be described in succession.

In the first mode, pressure for effecting the alternate dispersions is derived entirely from the feed pumps. The valves **43**, **100**, **101** and **102** are open but the surge pumps **34** and **37** are not used; they may be removed or locked against reciprocation. In the first half-cycle of the operational cycle the light-liquid valves **28** and **31** are open and the heavy-liquid valves **27** and **32** are closed; the piston **76** is at the left, whereby the contacting zone F is at minimum effective volume. A measured amount V_1 of lighter liquid, preferably less than the volume of light liquid contained in one contacting zone, is admitted under pressure by pump **74** to the bottom settling zone G, the quantity being measured, for example, by reference to the change in level in the measuring tank **73**. For example, this tank may be tall and of small cross section

and be filled from the conduit 71 to a first predetermined level, and thereafter liquid may be withdrawn by the pump 70 to a second predetermined level; this may be done automatically by providing level responsive devices (not shown, but known per se) for closing the valve 72 when the first predetermined level is reached and stopping the pump when the second predetermined level is reached. A volume V_1 of light phase is thereby forced through openings in the lowermost dispersing plate (the part of the plate 45 between zones F and G) into a continuous layer of heavy liquid phase in the adjoining contacting zone F, and light phase is formed from said zone F through the next plate and is dispersed into the layer of heavy liquid in the adjoining zone D. This action is repeated in each zone, whereby a dispersion of light liquid phase in heavy liquid phase is formed next to each perforated plate and previously settled light phase is forced from the uppermost settling zone A and discharged through the conduit 29.

During the second half-cycle, the valves 28 and 31 are closed and valves 27 and 32 are open, and a volume V_2 of the heavier liquid is delivered to the uppermost zone A by a pump 67 and measured in the tank 68, this quantity V_2 being preferably less than the volume of heavy liquid contained in one contacting zone. This volume may be selected independently of V_1; thus, it may be equal to, greater than, or less than V_1. This flow causes a volume V_2 of the heavier liquid to be dispersed from the bottom of each zone (except G) through the openings 46 of the partition into the body of lighter liquid in the upper part of the adjoining, lower zone, and previously settled heavy phase is forced from the lowermost settling zone G and discharged through the conduit 30.

When the interface level between the bodies of lighter and heavier liquid phases rises due to an increase in the volume of the solvent that constitutes the heavier liquid phase, corrective transport of heavier liquid to a higher compartment occurs during the dispersion of lighter liquid into the higher compartments, as was described in detail earlier herein. In this embodiment, however, a further compensating mechanism, viz., the piston 76, is employed. During the first half-cycle described above, the pressure in the uppermost zone A and, therefore, that in the space 98, are less than that in the zone F; hence, the floating piston 76 moves toward the right until stopped by the adjustable stop 78 to increase the capacity or effective volume of the zone F. As a result the quantity of light liquid that is dispersed from the zone F into the zone D is less than the quantity of light liquid that is dispersed into the zone F from the zone G. During the second half-cycle, the pressure in the zone A and, therefore, that in the space 98 are greater than that in the zone F; hence the piston 76 moves toward the left until stopped by the fixed stop 77 to decrease the capacity or effective volume of the zone F. As a result the quantity of heavy liquid that is dispersed from the zone F into the zone G exceeds the quantity that is dispersed into the zone F from the zone D. This alternate expansion and contraction of the effective volume of the zone F and the resultant changes in the flow quantities compensate for the decrease in the volume of the light liquid flowing through the zone F.

The extent of the change in effective volume during each half-cycle may be made such that the tendency for a shift in the interface level is exactly compensated for. Theoretically, the position of the stop 78 can be determined by calculations, or by trial and error; however, it is usually more expeditious to rely upon the action of the float 83. For a detailed discussion of the optimum changes in effective volume, reference is made to our aforesaid copending application, Serial No. 255,692, now Pat. No. 2,729,550, wherein alternate embodiments of the apparatus are also described. Since, however, in the apparatus of the instant invention there is the further corrective action due to transport of the liquid that tends to increase in volume, it is possible to adjust the stop 78 manually and clamp it by the screw 79 at a position to give a travel to the piston 76 that is suitable under most conditions to produce complete compensation, and then rely upon the transport of liquid to take care of minor corrections due to fluctuations in level. It is seen, therefore, that by combining the two ideas it becomes possible to simplify the construction by eliminating the float and linkage mechanism.

The apparatus according to Figure 16 may also be operated in other ways. For example, the surge pump 34 or 37 may be used to actuate the piston 76 by closing the valves 101 and 102. Considering further the system of liquids described above, wherein the heavier liquid increases in volume, valve 44 remains closed and valve 43 remains open, and surge pump 37 is not used. The feed pumps 67 and 74 and the valves in the feed and discharge conduits are operated exactly as described above to cause flow of liquid through the perforated partitions in alternate directions. The surge pump 34 is reciprocated to make a stroke to the left during each first half-cycle (while lighter liquid is introduced at the bottom and dispersed) and to make a stroke to the right during each second half-cycle. It is evident that this causes the pressure in the space 98 to be alternately decreased and increased to cause the same reciprocations of the piston 76 and the same alternate expansion and contraction of the effective volume of the zone F as were described above.

According to still another mode of operation, the valves 101 and 102 are open and both surge pumps 34 and 37 are used to cause the movement of liquids through the perforated partitions in alternate directions, as described previously for Figures 1 and 2; the feed pumps 67 and 74 are in this case operated to supply the feed liquids continuously or intermittently as also described for Figures 1 and 2. The piston 76 is actuated by keeping the valves 43 and 100 open and the valve 44 closed, to cause alternating pressures to be established in the space 98 in the manner described in the preceding paragraph.

It is evident that in any of the modes of operation described, the valve 43 would be closed and the valve 44 open when the lighter liquid increases in volume relatively to the heavier liquid. The movements of the piston 76 will thereby be opposite to those described above.

It will be further understood that various modifications may be made on the controls themselves without changing the principles involved. For example, the piston 76 may be actuated by cams instead of the hydraulic pressure. Likewise, the piston and cylinder may be replaced by a suitable diaphragm, metallic bellows, plunger that extends into the compartment F to occupy a variable space, etc. The control piston may also be spring- or pneumatically-loaded so that it stores up sufficient energy on the suction stroke to provide for the discharge. Moreover, it is possible to equip only some chambers with the perforated plates having passageways connecting points at different elevations for automatic transport of the liquid that tends to increase in volume and to construct one or more other chambers as variable-volume chambers; the latter may then have horizontal perforated partitions with all perforations at a common level, as described in the aforesaid copending application Serial No. 255,692 now Patent No. 2,729,550.

We claim as our invention:

1. In a reciprocal dispersion process, wherein pairs of continuous contacting bodies of different liquids A and B are established as layers within confined contact zones arranged as a series, each said zone being separated from the adjacent zone by a partition means providing a plurality of restricted flow openings, and the liquid A from each zone except the first in the series is dispersed repeatedly into the body of the liquid B in the adjoining zone toward the first end of the series by

passage through said flow openings, in alternation with repeated dispersals of the liquid B from each zone except the last in the series into the liquid A in the adjoining zone toward the other end of the series by passage through the same flow openings, and wherein said liquids A and B are flowed progressively through the zones with the liquid B is flowing from the first end to the said other end and the liquid A flowing in the opposite direction, the improvement which comprises correcting for a diminution in the depth of the contacting body of one liquid and the corresponding increase in the depth of the contacting body of the other liquid within the same contacting zone by transporting a part of the latter liquid from said zone through a fractional portion of said flow openings only during the dispersal of said one liquid into the adjoining zone in the direction toward which the said one liquid is dispersed.

2. Method of contacting at least partially immiscible liquids having relatively different densities the first of which liquids increases in volume relatively to the second liquid upon being contacted therewith, which comprises the steps of establishing a succession of contacting bodies of said first liquid alternating with contacting bodies of said second liquid, there being one continuous body of each of said liquids within each zone of a series of confined contacting zones as superposed layers in direct contact with each other; alternately (*a*) dispersing first liquid from the body of first liquid in each zone except the first in the series as a plurality of fine streams into an adjoining body of second liquid in an adjacent contacting zone toward the first end of said series and (*b*) dispersing second liquid from the body of second liquid in each zone except the last in the series as a plurality of fine streams into an adjoining body of first liquid in an adjacent contacting zone toward the other end of said series; and maintaining the depths of said superposed layers by transferring first liquid from at least one contacting body thereof into a contacting body of first liquid in an adjacent contacting zone toward said other end of the series only during the said dispersion step (*b*).

3. In combination with the method according to claim 2, the step of decreasing the effective volume of at least one of said confined contacting zones during the dispersion step (*a*) and increasing the effective volume of said one zone during the dispersion step (*b*).

4. Method of contacting at least partially immiscible liquids having relatively different densities the first of which liquids increases in volume relatively to the second liquid upon being contacted therewith, which comprises the steps of establishing a succession of contacting bodies of said first liquid at progressively different levels alternating with contacting bodies of said second liquid, there being one continuous body of each of said liquids within each zone of a series of confined contacting zones as superposed layers in direct contact with each other and separated from each other by a liquid-liquid interface, said bodies being situated so that the bottom of the lower liquid body within each zone except the lowermost zone is below the top of the upper liquid body in the adjacent lower zone, each said lower liquid body being separated from the said upper liquid body in the adjacent zone by a partition and being in communication therewith through a plurality of flow openings distributed through a range of levels; alternately (*a*) dispersing first liquid from the body of first liquid in each zone except the first in the series through said openings into an adjoining body of second liquid in an adjacent contacting zone toward the first end of said series and (*b*) dispersing second liquid from the body of second liquid in each zone except the last in the series through said openings into an adjoining body of first liquid in an adjacent contacting zone toward the other end of said series; and maintaining the depths of said superposed layers by transferring first liquid from at least one contacting body thereof into a contacting body of first liquid in an adjacent contacting zone toward said other end of the series simultaneously with the said dispersion step (*b*) whenever the said interface in said one contacting zone is at a level within said range of levels.

5. Apparatus for intimately contacting at least partially immiscible liquids having different densities by reciprocal dispersion comprising, in combination: a series of more than two adjoining enclosed chambers having intervening partitions, each of said partitions having a plurality of simultaneously open, restricted flow passageways extending through said partitions and interconnecting said chambers serially, the flow passageways through at least one partition opening into the chamber adjoining one side thereof directly at a plurality of levels within the adjoining chamber; a pair of feed conduits for the liquids to be contacted communicating with chambers respectively at the ends of the series; a pair of withdrawal conduits for the contacted liquids communicating with chambers respectively at the ends of the series; and means for causing movements of the liquids in said chambers through said passageways alternately in opposite directions.

6. Apparatus according to claim 5 wherein the passageways through said one partition open into the chamber adjoining the other side of said partition directly at a plurality of levels within said last-mentioned chamber.

7. Apparatus according to claim 5 wherein said intervening partition extends through different elevations and said passageways are orificial openings therein situated at progressively different levels.

8. Apparatus according to claim 7 wherein said intervening partition is substantially vertical.

9. Apparatus according to claim 7 wherein said intervening partition is inclined between the vertical and the horizontal.

10. Apparatus according to claim 5 wherein said intervening partition is horizontal and said passageways include a plurality of orificial openings through the partition and at least one tube extending from said partition into said adjoining chamber and in flow communication with said adjoining chamber at a level spaced from the partition and with the chamber adjoining the other side of the partition substantially at the level of the partition.

11. In combination with the apparatus according to claim 5, means for alternately increasing and decreasing volume of at least one of said chamber intermediate the ends of the series.

12. Apparatus for intimately contacting at least partially immiscible liquids having different densities by reciprocal dispersion comprising, in combination: an upright vessel; a wall structure within said vessel subdividing the vessel into a series of confined contacting chambers situated at progressively different elevations, said wall structure including partition walls that are situated between consecutive chambers and extend through different elevations, whereby the bottom of the higher chamber of the pair of chambers separated thereby is below the top of the lower chamber of said pair, each of said partition walls having a plurality of simultaneously open, orificial openings situated through a range of levels therein and opening into the chambers adjoining the partitions directly at a plurality of levels within the said chambers; a pair of liquid feed and liquid draw-off conduits communicating with said vessel at each end thereof; and means for causing flow of liquids through said orificial openings alternately in opposite directions.

13. Apparatus according to claim 12 wherein said partition walls are vertical and in the form of tubes situated within said vessel and spaced from the vessel wall, and said wall structure further includes imperforate walls closing the ends of the said tubes and additional imperforate walls extending from said tubes outwardly to the vessel wall, the said confined contacting chambers

being alternately within said tubes and outside the tubes.

14. Apparatus according to claim 12 wherein said partition walls are inclined between the vertical and the horizontal and consecutive partitions are substantially parallel.

15. Apparatus according to claim 12 wherein said partition walls are inclined between the vertical and the horizontal and consecutive partitions are inclined alternately toward opposite sides of the vessel.

16. In combination with the apparatus according to claim 12, means for alternately increasing and decreasing the effective volume of at least one chamber intermediate the top and bottom of said vessel synchronously with said alternate flow of liquids through the orificial openings.

17. As a subcombination, a contacting assembly for use in reciprocal dispersion column, including an upright tube closed at the vertical extremities and provided with a plurality of orificial openings distributed through the height thereof; and an imperforate partition extending laterally from the tube at a height intermediate the said extremities thereof and adapted to form a transverse partition within an upright vessel, both the portion of the

tube above the partition and the part below the partition having some of the said openings situated at progressively different levels.

18. Apparatus for intimately contacting at least partially immiscible liquids having different densities comprising, in combination: an upright vessel; a wall structure within said vessel including a plurality of tubes situated side by side at substantially a common level and closed at their top and bottom extremities and an imperforate partition situated outside of said tubes at a level intermediate the said extremities thereof and sealed to said tubes and to the inner wall of the vessel, said tubes having orificial openings distributed through a range of levels situated above said imperforate partition and orificial openings distributed through a range of levels situated beneath said imperforate partition.

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